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DETERMINATION OF SOLIDS CONTENT OF CHARCOAL-IMPREGNATED POLYURETHANE FOAMS USING DENSITY MEASUREMENTS

by

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ABSTRACT

A method to measure the density of charcoal-impregnated polyurethane foam which gives results with standard deviations of less than 0.7% has been developed. Curves relating density of impregnated foam to weight fraction of solids in the foam are presented. These curves allow the determination of the solids content of a foam sample by measuring its density, provided the density of the unimpregnated foam is known. If the composition of the impregnite is known, a more accurate estimation of the weight fraction of solids is possible and the charcoal content may also be determined.

RÉSUMÉ

On a mis au point une technique de mesure de la masse volumique de la mousse de polyuréthane imprégnée au charbon de bois donnant des résultats dont les écarts types sont inférieurs à 0,7 %. Sont présentées des courbes liant la masse volumique de la mousse imprégnée à la masse fractionnaire des solides présents dans la mousse. Ces courbes permettent de déterminer la teneur en solides de la mousse par mesure de sa masse volumique, pourvu que la masse volumique de la mousse non imprégnée soit connue. Lorsque la composition de l'imprégnant est connue, il est aussi possible de déterminer avec davantage de précision la masse fractionnaire des solides, ainsi que la teneur en charbon de bois.

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INTRODUCTION

The Canadian Chemical Warfare (CW) Protective Suit is comprised of two layers, an outer, nylon/cotton, repellent layer and an inner layer of polyurethane foam laminated to a nylon-tricot backing impregnated with charcoal. In addition to charcoal, the impregnating bath is likely to contain a latex binder, suspending agents, flame retardants, etc.

There are several tests to evaluate the inner layer. One of these, the carbon tetrachloride (CCl_4) adsorption test (1), measures the capacity of the impregnated foam to adsorb CCl_4. This test can be done before and after a sample is laundered to determine the effect of washing the material or before and after wear trials. Unfortunately, this test alone cannot allow us to differentiate between loss of adsorption capacity due to loss of charcoal and loss due to poisoning of the charcoal by the detergent or soilant.

A test method to determine the actual charcoal content has been developed. This method is to measure the density of the impregnated material. Knowing the density of both the unimpregnated foam and the impregnating solids (impregnite), the weight fraction of impregnite in the material can be calculated. The charcoal content can then be calculated from the charcoal concentration of the impregnite. Density measurements of fabrics with known amounts of impregnite were used to estimate an apparent or effective density of the impregnating solids.

This method was used earlier by Hart to estimate the charcoal lost during wear trials but no details were given on how density was measured or on how solids density was determined.

THEORY

The density of a porous solid such as polyurethane foam can be determined by weighing it in air and then immersed in a liquid which penetrates the pores. Density is calculated from

$$\rho_R = \frac{W_A \rho_L}{W_A - W_L} \qquad [1]$$

where W_L = weight of foam in liquid,

W_A = weight of foam in air,

ρ_L = density of liquid,

ρ_R = density of sample.

The weight of foam in air, W_A, and the density of the liquid, ρ_L, in which the foam is to be weighed are easily obtained. The weight of the foam in the liquid, W_L, is more difficult to determine because the foam must be completely wetted by the liquid leaving no air bubbles to affect the measured weight.

The charcoal impregnated foam can be thought of as a mixture of two solids with different densities, the polyurethane foam laminate and the charcoal-containing impregnite. Using the density of mixtures equation,

$$\rho_{AV} = \frac{\Sigma W_i}{\Sigma V_i} = \frac{\Sigma W_i}{\Sigma (W_i/\rho_i)} \qquad [2]$$

where ρ_{AV} = average density of the mixture

W_i = weight of i^{th} component

V_i = volume of i^{th} component

ρ_i = density of i^{th} component

and letting W_s = weight fraction of impregnated solids

$1-W_s$ = weight fraction of foam laminate

then from [2]

$$\rho_R = \frac{1}{\frac{W_s}{\rho_s} + \frac{1-W_s}{\rho_{FL}}} \qquad [3]$$

where ρ_{FL} = density of unimpregnated foam laminate

ρ_s = density of solids impregnated into foam laminate

Rearranging

$$W_s = \frac{1 - \rho_{FL}/\rho_R}{1 - \rho_{FL}/\rho_s} \qquad [4]$$

The density ρ_s can be calculated by applying the density of mixtures equation [2] to the components of the charcoal bath. To do this, the individual densities must be known and it must be assumed that these values are still correct after mixing the ingredients, impregnating the fabric and drying it. It is likely that this assumption is not valid for the latex binder which changes during drying from a suspension of particles to a bonding matrix nor for the charcoal because some of the pores may no longer be available to the immersing liquid.

An apparent solids density can be determined experimentally for known values of W_s using a rearrangement of equation [3]

$$\rho_s = \frac{W_s \rho_{FL} \rho_R}{W_s \rho_R + \rho_{FL} - \rho_R} \qquad [5]$$

For a series of experiments with the same foam laminate and charcoal impregnite bath formula the values of ρ_{FL} and ρ_s are constant. Solids fraction, W_s, is then related to ρ_R by an equation of the form

$$W_s = a - \frac{b}{\rho_R} \qquad [6]$$

where $a = \dfrac{\rho_s}{\rho_s - \rho_{FL}}$ and $b = \dfrac{\rho_s \rho_{FL}}{\rho_s - \rho_{FL}}$.

This equation can be used to draw a curve showing the relationship between W_s and ρ_R for a given foam laminate/charcoal bath system. It can also be used directly to determine the weight fraction of an impregnated sample after the experimental determination of ρ_R.

PROCEDURE

A specimen 2 inches in diameter was dried and weighed, then immersed in boiling water and held beneath a magnetic stirring bar. The water was allowed to cool while the stirring bar rotated on top of the foam. After 15 minutes the sample was quickly transferred to room-temperature distilled water. A small wire hook was suspended from the weighing apparatus with enough of the hook below the water that the specimen could be suspended completely submerged. After the hook was weighed alone, the circle was pierced by the hook while it was still submerged and then they were weighed together. Using equation [1], the density was calculated (see Appendix A). Three replicates were made per sample to give a final average density.

A wetting agent (3 drops of a 5% solution of Triton X102) was used in the boiling water to aid in wetting of the samples. Subsequent testing revealed that this made no significant difference.

Liquids with low surface tension such as hexane and ethanol were used at room temperature to try to wet the circles. These attempts were unsuccessful.

Density determinations were performed on 17 samples of charcoal impregnated polyurethane foam with the regular nylon tricot backing on one side. Seven of these had been impregnated with a charcoal mixture without flame retardant while the other ten had a flame retardant additive. There were also six samples with light-weight nylon tricot on both sides of the polyurethane foam impregnated with charcoal mixtures containing a flame retardant. Densities were also determined for nine samples of unimpregnated foam laminate.

Four charcoal-impregnated foam-laminates W, X, Y and Z were tested to determine the solids fraction by measuring their density. They were sampled every 4.3 m, giving three samples of Y (double-sided foam laminate with flame retardant) and Z (single-sided foam laminate without flame retardant) and six samples of W and X (both single-sided foam laminate with flame retardant). At each sampling point, three replicate density measurements were made. Averages were taken to give an overall density of each of W, X, Y and Z. From these density values, the solids weight fraction, W_s, was calculated (equation [4]) using an experimentally determined value of ρ_{FL} and two different values of solids density. The results were then compared to the experimental add-on measured during the impregnation of the foam laminate. One solids density was the theoretical density calculated from component densities in the charcoal bath using equation [2]. The other solids density was the average apparent density (equation [5]) for the appropriate impregnated foam laminate taken from the 17 samples referred to earlier.

The theoretical densities of charcoal bath solids were calculated using the density of mixtures equation [2] and individual densities of the components. The component densities were collected from various sources including manufacturers' bulletins and handbooks. The charcoal densities used were taken from pycnometer density measurements.

RESULTS AND DISCUSSION

Table I lists experimental densities of the unimpregnated foam laminates.

Table II lists the results of density determinations of the single-sided material without flame retardant. Good repeatability is shown with standard deviation for each material (n=3) ranging from 0.8 kg/m^3 to 6.4 kg/m^3 and averaging 2.9 kg/m^3 which is 0.22% of the average value of ρ_R. Table III shows the experimental densities of the single-sided, flame-retardant foam. Again, good repeatability is shown by the standard deviations ranging from 0.4 kg/m^3 to 8.7 kg/m^3 and averaging 3.9 kg/m^3 or 0.27% of the mean. Table IV shows the densities of the double-sided flame-retardant foam laminate. The standard deviations ranged from 1.7 kg/m^3 to 9.7 kg/m^3 and averaged 6.2 kg/m^3 or 0.43% of the mean. The largest standard deviation expressed as a percentage of its corresponding ρ_R was less than 0.7%, indicating high precision in the measurement of density.

Experimental solids densities (ρ_S) were calculated from equation [5] for each padding run and are given in Tables II, III and IV. The average from each table is listed in Table V along with theoretical densities as a comparison. The difference between the theoretical and the experimental values could be due to inaccuracies in densities for the individual components from which the theoretical values are calculated. As explained earlier, there is also the effect of drying the latex and the effect of this on the pores of the charcoal. Any air trapped in pores would contribute a buoyancy effect making the experimental density less than the theoretical density. As would be expected there was no significant difference (P=99.9%) between solids densities for one-sided foam with flame retardant and two-sided foam with flame retardant according to a t-test on both theoretical and experimental densities.

Once the experimental densities were determined, curves of solids fraction (W_S) versus impregnated foam density (ρ_R) were drawn for the three experimental conditions: single-sided foam with and without flame retardant and double-sided foam with flame retardant. The average experimental density from Table V and the average ρ_{FL} for each experimental condition, were substituted into equation [6] to give the three curves shown in Figure 1. Although there were essentially only two charcoal bath formulas (with or without flame retardant), there were some small differences which could have

TABLE I

Density of Unimpregnated Foam Laminate

Foam Laminate		From Run	Density ρ_{FL} (kg/m^3)	Standard Deviation (kg/m^3)
Single Sided	35R1	436	1209.7	± 3.0
	34C	395	1203.2	± 3.1
	34C	383	1206.3	± 1.9
	34C	386	1207.2	± 1.7
	34D	390	1209.2	± 1.2
	35C	398	1207.3	± 1.2
	35D	412	1208.2	± 0.9
Double Sided	48L2	420	1196.8	± 1.5
	48L2	430	1191.3	± 7.5

TABLE II

Single-Sided Foam Laminate Without Flame Retardant

Run	Foam Laminate	Exptl W_s	ρ_{FL} (kg/m^3)	ρ_R (kg/m^3)		ρ_s (kg/m^3) (from eqn [5])
				Average	Std. Dev.	
396	35C	0.118	1207.3	1253.6	± 1.5	1757.3
398	35C	0.419	1207.3	1360.6	± 4.5	1652.8
412	35D	0.090	1208.2	1229.3	± 4.5	1492.9
417	35D	0.456	1208.2	1373.2	± 0.8	1640.5
383	34C	0.441	1205.6	1405.8	± 1.2	1780.6
395	34C	0.161	1205.6	1262.7	± 1.3	1676.5
391	34D	0.434	1209.2	1369.6	± 6.4	1656.1

Average ρ_s = 1665.2 kg/m^3; 95% confidence limits = ± 86.5 kg/m^3

TABLE III

Single-Sided Foam Laminate with Flame-Retardant Formula

Run	Foam Laminate	Exptl W_s	ρ_{FL} (kg/m^3)	ρ_R(kg/m^3)		ρ_s(kg/m^3) (from eqn [5])
				Average	Std. Dev.	
385	34C	0.541	1205.6	1507.0	± 0.4	1912.7
386	34C	0.523	1205.6	1477.0	± 3.7	1858.6
390	34D	0.532	1209.2	1477.2	± 3.5	1835.0
397	35C	0.506	1207.3	1478.4	± 8.2	1893.5
425	35R1	0.470	1209.7	1451.2	± 4.7	1872.8
431	35R1	0.522	1209.7	1468.9	± 5.7	1827.5
432	35R1	0.532	1209.7	1489.2	± 8.7	1869.1
435	35R1	0.496	1209.7	1468.7	± 3.3	1877.1
436	35R1	0.458	1209.7	1433.9	± 0.9	1836.7
393	35C	0.485	1207.3	1433.8	± 0.5	1790.5

Average ρ_s = 1857.4 kg/m^3; 95% confidence limits = ± 25.5 kg/m^3.

TABLE IV

Double-Sided Foam Laminate with Flame-Retardant Formula

Run	Foam Laminate	Exptl W_s	ρ_{FL} (kg/m^3)	ρ_R(kg/m^3)		ρ_s(kg/m^3) (from eqn [5])
				Average	Std. Dev.	
429	48L2	0.473	1196.8	1442.1	± 1.7	1868.9
420	48L2	0.418	1191.3	1369.6	± 7.1	1730.1
439	48L2	0.528	1196.8	1479.2	± 3.6	1874.6
440	48L2	0.487	1196.8	1453.4	± 9.4	1877.4
430	48L2	0.491	1196.8	1447.2	± 9.7	1848.0
423	48L2	0.505	1196.8	1480.8	± 5.5	1929.6

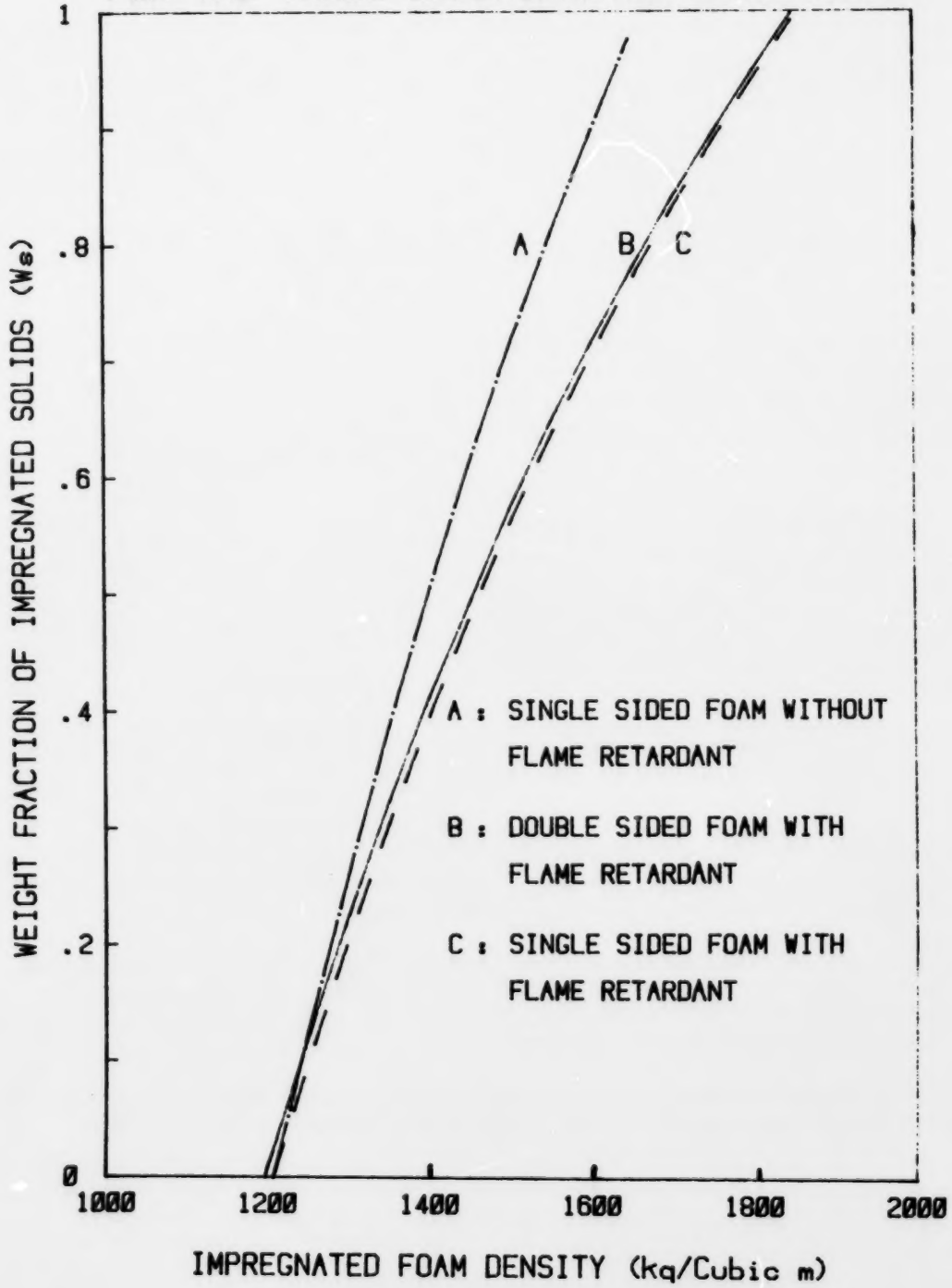
Average ρ_s = 1852.1 kg/m^3; 95% confidence limits = ± 76.4 kg/m^3.

TABLE V

Comparison of Theoretical and Experimental Solids Densities

	Theoretical ρ_s (kg/m^3)	Experimental ρ_s (kg/m^3)	Percent Difference
One-sided foam and no flame retardant	1732.7	1665.2	-3.90
One-sided foam and flame retardant	1919.2	1857.4	-3.22
Two-sided foam and flame retardant	1925.7	1852.1	-3.82

FIGURE 1 : COMPARISON OF DENSITY CURVES FOR THE THREE EXPERIMENTAL CONDITIONS



affected the solids density. The slight difference between curves B and C could also be attributed to the different foam laminates.

Table VI shows a comparison between the two methods used to calculate the solids fraction from the density measurements made on charcoal impregnated foam samples W, X, Y and Z. The padding value for W_s is an average over the entire padding run. Variations in solids add-on would lead to variations in solids fraction and density. It appears from Table VI that it is more accurate to calculate ρ_s on an individual basis using components of the charcoal bath rather than using an apparent or experimental ρ_s. However, in the event that the exact formula of the charcoal bath is not known, the use of the experimental ρ_s results in a reasonable approximation.

After the density determination had been made on the samples, they were then dried and weighed again to observe what effect the test had on the samples. Samples that had been impregnated with charcoal showed a small increase in weight. This was probably due to the charcoal adsorbing the wetting agent. The unimpregnated foam samples showed a small decrease in weight probably as a result of the rubbing action of the stirring bar on the sample. Both the increase and decrease in weight represented approximately 0.5% change.

CONCLUSIONS

1. The method to determine the density of charcoal-impregnated foam laminate gave standard deviations of less than 0.7%.

2. It is more accurate to determine the weight fraction of solids with a solids density calculated using the charcoal-bath formula than with an apparent density taken from the curves. The apparent density, however, does give a good approximation should the formula be unknown.

3. By determining the charcoal content using the methods outlined in this report, a differentiation may now be made between the loss of adsorption capacity due to loss of charcoal and loss due to poisoning of the charcoal by the detergent or soilant.

4. A wetting agent is not necessary to provide consistent or optimum wetting of the impregnated foam if the proper procedure is followed.

TABLE VI

Comparison of Two Methods of Calculation of Solids Fraction with Solids Fraction from Padding

Sample	Padding W_s	Using Individual ρ_s from Bath Formula		Using Experimental ρ_s from Table V	
		W_s	% difference from padding W_s	W_s	% difference from padding W_s
W	0.529	0.538	+1.7	0.549	+3.8
X	0.505	0.544	+7.7	0.544	+7.7
Y	0.458	0.470	+2.6	0.489	+6.8
Z	0.424	0.411	-3.1	0.434	+2.4

REFERENCES

1. Canadian Forces Specification D-80-001-037/SF-001: Cloth, Laminated Nylon and Polyurethane Foam, Impregnated, Test Method 4.4.1, Adsorption Activity, 30 May 1980.

NOTATION

$a = \rho_s/(\rho_s - \rho_{FL})$

$b = \rho_s\rho_{FL}/(\rho_s - \rho_{FL})$

W_A = weight in air

W_L = weight in liquid

W_s = weight fraction of solids

V = volume

ρ_{AV} = average density

ρ_{FL} = density of unimpregnated foam laminate

ρ_L = density of liquid

ρ_R = measured density of charcoal-impregnated foam laminate

ρ_s = density of solids in mixture impregnated into foam laminate

APPENDIX A

EXAMPLE CALCULATIONS

RUN 436 - Single-sided foam laminate, flame-retardant formula.

1) To find the density of impregnated foam laminate (ρ_R) using equation [1].

Replicate #	Dried Weight W_A (g)	Weight of Hook (g)	Hook & Wetted Foam (g)	W_L	Density ρ_R(kg/m^3)
1	0.44278	0.09335	0.22787	0.13452	1433.1
2	0.44178	0.09285	0.22722	0.13437	1433.8
3	0.44453	0.09322	0.22863	0.13541	1434.8

ρ_L = density of water at ambient temperature (22.2°C) = 997.72 kg/m^3.*

Average of three replicates = 1433.9 kg/m^3. Standard deviation = 0.9 kg/m^3

* Handbook of Chemistry and Physics, 48th Edition, 1967-8, page F-4.

2) To calculate the apparent density of the solids add-on (ρ_s) using equation [5].

Run #	Foam Laminate	ρ_{FL} (kg/m^3)	ρ_R (kg/m^3)	W_s
436	35R1	1209.7	1433.9	0.458

ρ_s = 1836.7 kg/m^3.

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